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RESEARCH AND DEVELOPMENT PROGRAM

MONTHLY PROGRESS REPORT NUMBER 4
Period 1 September 1963 to 1 October 1963
Contract Number NAS 8-5438
Request Number TP 3-83547

prepared for
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Huntsville, Alabama

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ABSTRACT

This report covers the period 1 September 1963 to 1 October 1963, under Contract NAS 8-5438, which calls for twelve months of research and development of a high temperature thermocouple capable of measuring rocket engine exhaust temperatures in the 3000°C range, under adverse conditions of oxidation, erosion, vibration and shock.

The primary objectives of the program are to advance the state-of-the-art of high temperature thermometry, and to develop an end product suitable for in-flight temperature measurements on the SATURN vehicle.

Piece parts for six prototypes have been fabricated. Three will be delivered on 17 October 1963 per schedule. Three will be subjected to physical and electrical tests at ACL. First calibration tests have produced output curves parallel to those previously taken by both N.A.S.A. and ACL up to 2200°F. Oxidation effects on one gauge over six temperature cycles with a total time of five hours were less than anticipated. Literature searches and investigative tests of high temperature materials were continued.

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SECTION I

SUMMARY

1.0 Period Covered

This report covers the period 1 September 1963 to 1 October 1963.

1.1 Statement of Work

The Contractor shall advance the state-of-the-art of high temperature thermometry and specifically improve the technique of accurately measuring high temperatures by designing, fabricating, testing, and delivering nine (9) thermocouple probes capable of operation in the 3000°C temperature range under adverse conditions of erosion, oxidations and high stress levels for useful periods of time. Also, present methods of thermocouple probe fabrication will be modified such that the end product will be suitable for in-flight temperature measurements on the SATURN vehicle.

To accomplish the above objectives, the Contractor shall consider and explore specific R&D efforts as follows:

a. Development of the physical structure of an immersed probe to attain minimum drag and highest resistance to bending and shear forces.

b. Ascertain the best combination of ingredients in the protective coating of the probe to extend the term of oxidation resistance.

c. Determine the best combination of compensated lead wires for use with immersion type probes.

d. Incorporate latest state-of-the-art materials as potting and sealing elements in the base of the probe.

1.1 Statement of Work Cont....

e. Determine effects of reactions between oxide coatings and tungsten in relation to the emf output.

f. Establishment of rates of erosion for different types of refractory coatings such as tungsten disilicide, carbides and cermets when subjected to high velocity, high temperature gas streams.

1.2 Progress

Accomplished during the current reporting period were:

a. Parts Fabrication:

Piece parts were fabricated for a total of six prototype gauges. It is planned to deliver three of the assembled gauges to N.A.S.A. on 17 October 1963 per schedule. The remaining three assemblies will be subjected to calibration and physical tests by ACL. Some changes in detail from the materials and processes planned for the first parts have been necessitated by lead times from vendors longer than scheduled.

b. Test Oven

A small "blackbody" oven, electrically heated, was constructed for use in tests in the 1000°F to 2200°F temperature range.

c. Calibrations

One gauge, nearly identical to those to be delivered, was subjected to calibrations in the test oven over the 1300°F to 2200°F range. In six temperature cycles, the output emf curves were essentially duplicated in each run. The curve, however was seen to shift higher than observed in previous calibrations. This shift is presently believed due to a greater immersion depth than that used in previous calibrations and the effect of a different kind of lead wire.

1.2 Progress Cont....

d. Oxidation Test

A Tungsten slug, formed by the vapor deposition process was subjected to an oxidation test over a period of two hours. Using the increase in weight method, the oxidation rate in air was estimated approximately at one gram per hour.

e. Emissivity Test

A sample of coated Tungsten sheath material was subjected to an emissivity test in the test oven, since there are no published data on the spectral emissivity of the protective coatings. Emissivity is estimated at 0.4.

f. Post Mortem Examination of Type 4734 Gauge

The two ACL (PDL) Type 4734 gauges previously tested by N.A.S.A. in a scale version of a rocket engine were disassembled and examined. Gauge, Serial No. 003, run at 10 diameters downstream failed due to melting away of the junction and tip, indicating that temperatures in excess of 6000°F were encountered.

Gauge, Serial No. 001, run at 40 diameters, then moved in to 20 diameters failed because of sheath breakage.

g. Data Accumulation

Files for data accumulation and reduction have been set up for the more rigorous work to be performed in the second four month period.

SECTION II

PAST PROGRESS

2.0 General

Previous effort was reported in ACL Progress Reports T-1097-1 through T-1097-3.

2.1 Prototype Design

As was previously reported, objectives for the first prototypes have been limited to the 4000°F - 4500°F range in the interest of accumulating test data for analysis, the results to be utilized in future design.

A design approach for the prototype gauges was selected, and drawings prepared, detailing means of fabrication and assembly.

Investigations made into fabrication techniques involved in working vapor deposited Tungsten, resulted in improved material handling techniques.

Shock and vibration tests, performed on a prototype mock-up, resulted in a conclusion that the sheath material was intrinsically capable of withstanding the specified shock and vibration requirements.

Samples of various types of compensated lead wires were ordered for test and evaluation. All samples were not received in time for test.

An evaluation of the SRI calibration tests for ACL Type 4734 gauges was made, resulting in a conclusion that an optimum immersion depth might be in the order of 1-1/2 inches in an isothermal region.

Preparations were made for examination of the two Type 4734 gauges tested by N.A.S.A.

A test of a "no-insulation" approach was started, but was aborted due to a failure in the test oven.

SECTION III

CURRENT PROGRESS

3.0 General

Design of the first set of prototypes was frozen during the current reporting period in order to permit meeting the 17 October 1963 date scheduled for delivery. Effort during the reporting period was thus confined principally to fabrication of piece parts and development of assembly techniques. Calibrations were also performed in the 1300°F to 3200°F portion of the operating range.

3.1 Progress

3.1.1 Description of Prototypes

Features of the ACL Type 4735 prototypes gauges are described as follows:

3.1.1.1 Sheath

The sheath is identical to that shown in Report T-1097-3, with .4 inches trimmed from the back end in order to shorten the probe body as much as possible. The length of the exposed portion of the sheath will be 1.8 inches.

3.1.1.2 Body

The body of the gauge will be shortened by .15 inches from that shown in Report T-1097-3 to reduce the overhang at the back end and minimize the reaction to dynamic loads.

3.1.1.3 Insulation, Electrical

Primary insulation will be hard-fired Beryllium oxide tubing. Secondary insulation will be compacted Magnesium oxide powder, between the inner wall of the sheath and the outside wall of the Beryllium oxide tube. Magnesium oxide powder will also be used to fill other voids within the body.

3.1.1.4 Oxidation Protection

The sheath of the probe will be treated with a thin coating of Tungsten Disilicide, with an overlay of Silicon. The exposed sheath near the mounting fitting will be coated with Dynatherm D-65 intumescent material, to afford both oxidation protection and thermal insulation near the base of the immersed portion of the gauge. To afford a maximum of protection to the internal parts of the gauge, it is planned to complete the final assembly in an atmosphere of Argon.

3.1.1.5 Lead Wires

As indicated in the discussion of the calibration runs, the lead wire investigations have not been completed. Samples of lead wire insulated with MgO and swaged in stainless steel tubing were late in delivery. Thus, it will not be possible to incorporate this material in the first deliveries. As an interim measure, the leads will be glass-insulated, and fed through a stainless steel tube, filled with Magnesium Oxide.

3.1.2 Fabrication

3.1.2.1 Transition Section

The transitions from the Tungsten sheath to the positive compensated lead will be made by nickel brazing in an inert atmosphere. This technique was chosen because 1) it is recommended by authorities* on the brazing of Tungsten, and 2) it was successfully employed by ACL in the Type 4734 gauges. Examination of the two Type 4734 gauges tested at N.A.S.A. revealed that the braze joints made with this technique were not affected by the tests.

*Fansteel Metallurgical Corp.
Chicago, Illinois
Tech. Bulletin 1.301

3.1.2.2 Body

The bodies will be assembled using torque values (developed by test) necessary to effect sealing against 2000 psig.

3.1.2.3 Sheath Installation

The sheath will be installed as shown in Figure 1. The platinum wire spiral will be welded to itself at two points; one at the front of the cone, the other at the back of the cone. Upon tightening, the platinum will deform to effect a seal between the sheath and the union in which it is mounted. Tests at ACL show that good alignment and centering results from this method. Platinum is used because of its good ductility, relatively high melting point, and resistance to oxidation.

3.1.2.4 Lead Wire

It had been planned to employ compensated lead wire, insulated with Magnesium Oxide, and swaged into a stainless steel sheath, for the first prototypes. This material was not received in time for fabrication of the first gauges. An alternative type of lead extension will, therefore, be employed. The individual compensated lead wires will be insulated with fiberglass and will be fed through a length of stainless steel tubing welded to the cap of the body assembly. A standard ACL seal will then be used at the end of the lead wire tube. The tubing can then be bent with an ordinary tubing bender to accomodate the installation. This type of lead wire arrangement has been tested at ACL and has been found to function within acceptable limits of accuracy. Future deliveries will, however, employ the swaged material.

3.1.2.5 Union Material

It was planned to fabricate the union, (P/N 4735-31-3) by which the gauge is mounted, from fully annealed Tantalum, because of its refractory characteristics, and compatibility with Tungsten and Platinum. Long lead times for the Tantalum material, and difficult machining problems precluded the possibility of using this approach in the first prototypes. Therefore, the union will be made of 300 Series stainless steel, as in the Type 4734 gauges.

3.1.2.5 Union Material Cont....

During examination of the two Type 4734 probes, neither of the stainless steel unions showed adverse effects from the tests, for the period of the runs.

3.1.3 Calibrations, 1300°F to 2200°F

A series of calibration runs were made to establish calibration of the Type 4735 gauges. These are presented in Figure 2. A schematic diagram of the test setup is shown below in Figure 3. During the runs, an attempt was also made to determine an emissivity correction for use of the optical pyrometer.

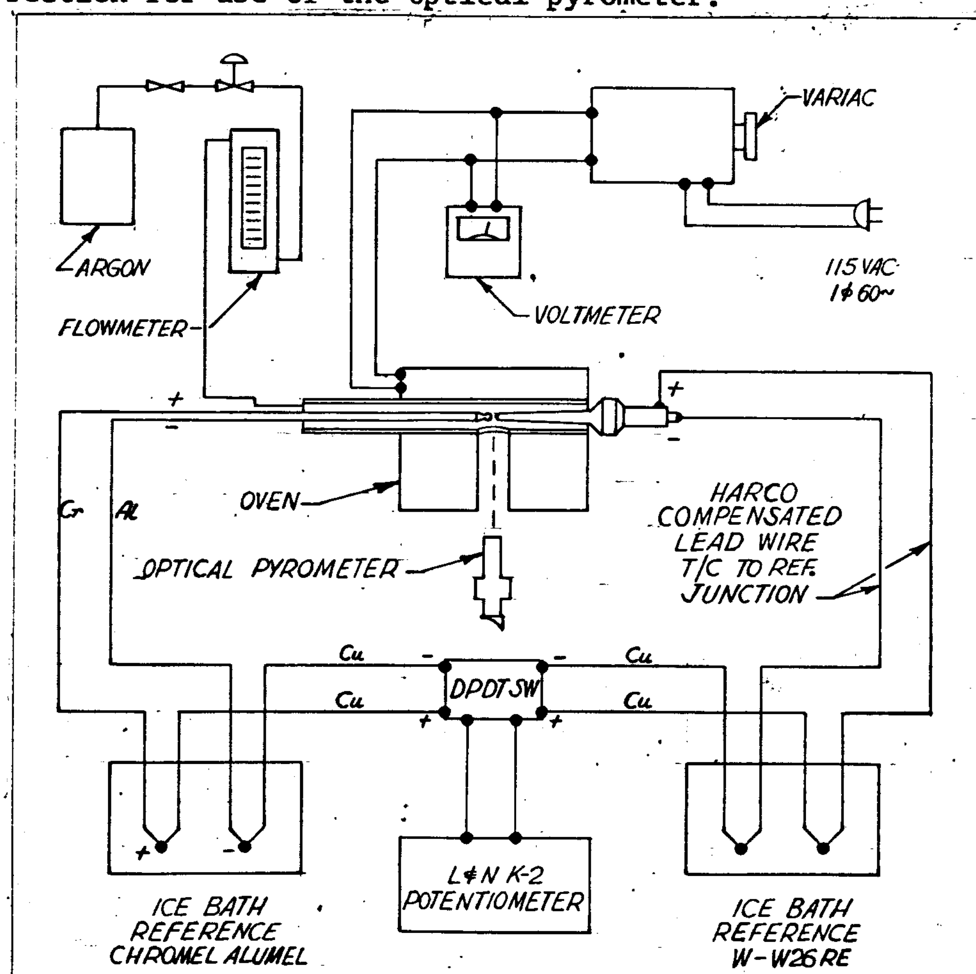

Figure 3
Calibration Test Setup

3.1.3.1 Run No. 1

Basic setup conditions for Run No. 1 were as follows:

Gauge Serial No.:	001
Immersion Depth:	2.3 inches
Atmosphere:	Argon
Temperature Range:	1300°F to 2300°F

This run was made without a reference thermocouple in the oven. All observations were made directly on the gauge tip, for a large number of points within the range. These data are shown in Figure 2 as circles (ascending) and filled circles (descending). The run showed excellent repeatibility and very low hysteresis.

Running time was 1 hour 10 minutes.

3.1.3.2 Run No. 1-1

Using the same setup as in Run No. 1, optical pyrometer readings were taken (ascending only) over the same temperature range. The data points are plotted as filled triangles in Figure 2. The curve was shifted slightly, but still showed good repeatibility within the range.

Running time was 30 minutes.

3.1.3.3 Run No. 2

Run No. 2 was aborted, after one data point was taken, when it was discovered that copper wire, rather than compensated lead wire, had been run to the reference junction. The single point was 6.2 mv at 700°C (1292°F) with a reference thermocouple in the oven.

Total running time was 30 minutes.

3.1.3.4 Run No. 3

Run No. 3 was made with a calibrated reference thermocouple in the oven, located in proximity to, but not touching, the 4735 gauge. The installation was made such that the pyrometer filament could be matched alternately with both thermocouple junctions.

Basic setup conditions for Run No. 3 were as follows:

Gauge Serial No.:	001
Immersion Depth:	2.0 inches
Atmosphere:	50% Argon - 50% Air
Temperature Range:	1300°F to 2100°F

At each stabilized temperature, at least two optical pyrometer readings were taken on both the reference thermocouple and the 4735 gauge. At the same time, the emf outputs of both thermocouples were recorded.

Ascending readings for the 4735 gauge are shown as open squares in Figure 2. Descending readings are shown as filled squares. As plotted the curve shows good repeatibility compared with Runs No. 1, and 1-1.

Total running time was 2 hours 27 minutes.

3.1.3.5 Discussion

When the curves developed during Runs No. 1, 1-1, and 3 are plotted as a composite curve to the same scale as the Southern Research Institute final calibration curve, (see Figure 4) it is seen that the curves taken by ACL are very closely parallel, although shifted to a higher emf value for the same temperature.

It should be noted that the SRI curves of immersion depth vs output were taken for the Type 4734 gauges at immersion depths of from 1/4" to 1-5/16". Their final calibration curve was apparently taken at 1-7/16 immersion depth, which was the maximum penetration possible with the SRI blackbody cavity. All of the SRI curves

3.1.3.5 Discussion Cont....

showed an increase in emf for the same temperature with increase in immersion depth, i.e. - a shift to the right. The ACL calibrations show a similar shift. If the shift per increment of immersion depth on the Type 4734 gauges is considered when comparing the two sets of curves, the emf shift in the Type 4735 gauges can be partly accounted for.

Other considerations which are under investigation as this report is in preparation are: 1) the effect of different compensated lead wire and 2) verification of blackbody conditions in the oven.

The effect of different lead wires is of interest because the Type 4734 gauges incorporated Minneapolis-Honeywell compensated lead wire (copper - copper nickel), whereas the ACL tests of the Type 4735 gauges incorporated Harco compensated lead wire. It is interesting to note that the single point taken in Run No. 2 fell on an extrapolation of the SRI curve downward to 1292°F. If it is found that there is, in fact, a series emf increase with the Harco lead wire, the difference is explained. If no such series effect is found, the difference may be attributed to non blackbody conditions at the time of observation, and an appropriate correction for emissivity must be applied to the pyrometer readings. Although Table I of this report seems to disprove this approach, such a correction would tend to shift the curve in the proper direction, i.e. - a higher temperature for the same emf output.

Thus far in the calibrations, it can be concluded that the Type 4735 gauges are capable of extended operation within the temperature range investigated, and the output of the gauge vs temperature is highly repeatable for a given thermal equilibrium condition. (Immersion depth and ambient temperature constant)

3.1.3.6 Oxidation Test

Concurrent with the calibrations of Para. 3.1.3 above, a quick look at oxidation characteristics of the vapor deposited Tungsten was taken. A Tungsten slug was weighed before and after a run in air.

3.1.3.6 Oxidation Test Cont....

Weight of the slug prior to the run was 9.34 grams, after a two hour run, the weight increased to 9.61 grams; an increase of .27 grams, or 2.87%. Assuming the increase in weight is attributed to the formation of $W_2 O_3$, a loss of the parent metal to the formation of the oxide of about one gram per hour was experienced.

Since the oxidation rate can be expected to increase rapidly with temperature, and additionally with the percent O_2, a conclusion can not be drawn at this time, as to the rate of oxidation in use.

Oxidation of the Type 4735 gauge was noticeable, but was not nearly as severe as expected.

3.1.4 Test Oven

Because of the well known difficulties in obtaining accurate temperature readings with an optical pyrometer, under conditions other than "blackbody" a small oven was constructed for use in calibrating. The oven was made per recommendations of the National Bureau of Standards*. It consists of an alumina tube fitted with a side port for viewing, surrounded by an electrical heater. The heater is helically wound over the alumina tube except in the region of the viewing port, where parallel windings were used. High temperature cement (Sauereisen No. 1) was used to coat the heater windings. The tubes were insulated with Fiberfrax, and the whole assembly was enclosed in a metal can.

The heater wire was Tophet A, 28 gauge, with a total resistance of 22 ohms. 115 VAC, 1 Ø, 60 cycle power was used, controlled with a 7.5 ampere variac. The voltage settings for approximate temperatures were determined with a calibrated chromel-alumel thermocouple, and a 9.34 gram Tungsten load in the oven. When temperature stability was reached, the voltage was recorded, and a number of readings were taken on the thermocouple junction with the optical pyrometer.

*NBS Monograph 41, Theory and Methods of Optical Pyrometry

3.1.4 Test Oven Cont....

Typical comparative readings are given in Table I below.

TABLE I

OVEN CALIBRATION

Calibration Temperature °F	Optical Pyrometer Reading	Calibration Temperature °F	Optical Pyrometer Reading
1513	1517	1965	1960
1513	1513	1980	1976
1729	1733	1980	1978
1745	1742	2146	2140
1960	1944	2148	2130

The readings listed in Table I represent the best and worst readings taken by both experienced and inexperienced personnel. Experienced individuals are able to repeat readings to a high degree. Individuals among them are capable of tracking the thermocouple readings within a few degrees F.

3.1.5 Post Mortem Examination, Type 4734 Gauges

The two Type 4734 gauges previously tested at N.A.S.A. in a scale rocket engine were disassembled and examined.

3.1.5 Post Mortem Examination, Type 4734 Gauges Cont....

Gauge Serial No. 003

This probe was run at 10 diameters downstream of the rocket nozzle. An open circuit was observed after 3 seconds. The examination of the part quickly revealed that the junction had been melted away, thus explaining the open circuit. The probe was complete disassembled, and the piece parts were examined.

The sheath was found to be severely eroded for about two inches from the deflection shield used for mounting, although the front portion was relatively undamaged except for the tip itself.

It is believed that the steel of the deflection shield flowed back up the sheath upon melting. Tungsten, being soluble in molten steel, went into solution as the steel flowed forward. Boundary layer theory of laminar separation would seem to support this belief*.

Internally, the braze joint between the copper conductor and the Tungsten sheath remained intact, as did the brazed joint between the Tungsten Rhenium center conductor and its cupro-nickel lead wire. In the region of the copper joint, the Beryllium oxide was intact, although it had melted near the tip. A teflon seal at the back end of the body of the gauge was undamaged. The stainless steel of the body was undamaged. All screw-joint parts functioned normally.

Gauge Serial No. 001

This probe was run at 40 diameters for 15 seconds, shut down and reinstalled at 20 diameters, then run for 10 seconds.

Upon receipt of the gauge the sheath was broken at a point just inside the mounting fitting. The break was typical of the type of break previously induced in the 4734 sheaths by a sharp-edged blow, or excessive shock. The broken faces of the sheath show evidence of oxidation, which indicates the break occurred during or prior to the run.

*High Speed Aerodynamics, Hilton, Longmans Green & Co., P.P. 587-588

3.1.5 Post Mortem Examination, Type 4734 Gauges Cont....

Gauge Serial No. 001 Cont....

The junction was found to be intact, as well as all other electrical connections.

The mounting employed in the Type 4735 gauge is designed to prevent the type of failure seen in this gauge.

3.1.6 Calibration, 3000°F

In order to establish a calibration point higher in temperature than those taken in the calibrations between 1300°F and 2300°F, the same gauge, Serial No. 001, was mounted in a steel deflection shield, and run in an oxy-acetylene burner. The point shown near 3000°F in Figure 4 is the average of twenty-eight readings. The large number of readings were taken because of fluctuations in temperature seen during the run. The highest temperature was 3182°F, the lowest 2948°F. Calibrations at higher temperatures will be made in the next reporting period.

SECTION IV

PROGRAM FOR NEXT INTERVAL

4.0 Objectives for the interval 1 October 1963 to 1 November 1963.

a. Continue literature search for high temperature insulators and coating materials.

b. Prepare three Type 4735 gauges for delivery to N.A.S.A. on 17 October 1963.

c. Prepare for visit to N.A.S.A. by ACL technical representative on 17 October 1963.

d. Continue calibrations.

e. Continue oxidation tests.

f. Continue lead wire tests.

g. Reduce data and analyze test results of first prototypes.

SECTION V

STATEMENT OF MAN HOURS

5.0 Hours by Category

Category	Previous Periods	Current Period	To Date
Engineering	276.5	100.0	376.5
Clerical	31.0	20.5	5.150
Fabrication	172.5	129.25	301.75
Consulting	-0-	-0-	-0-
Drafting	19.0	-0-	19.0

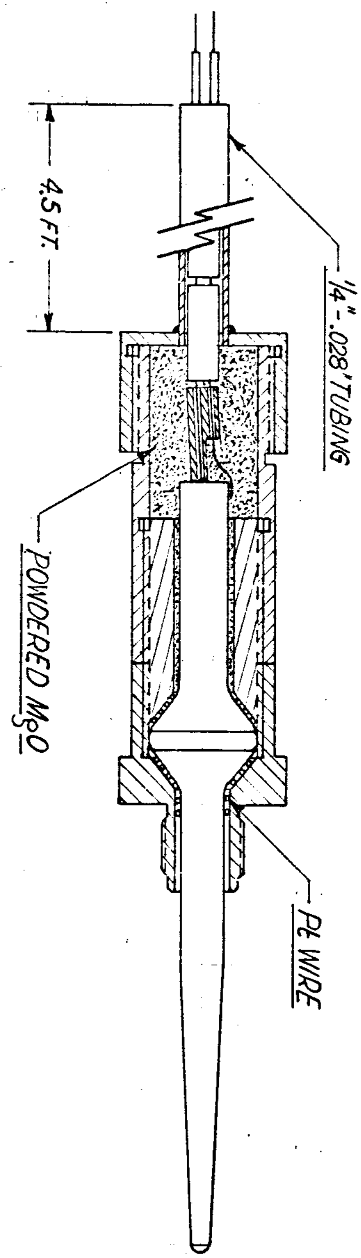

FIGURE 1

Assembly Sketch, ACL Gauge, Type 4735